

PATENT SPECIFICATION



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COMPLETE SPECIFICATION.

Improved Process for the Production of Pigments and Turbidity Agents.

We, DEUTSCHE GOLD- UND SILBER-SCHNEIDANSTALT, vormals Roessler, a corporation organised under the Laws of Germany, of 7/9, Weissfrauenstrasse, Frankfurt am Main, Germany, and LUDWIG WEISS, a German citizen, of 20, Mittelweg, Frankfurt am Main, Germany, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

The present invention relates to the manufacture of voluminous highly dispersive paint pigments and turbidity agents from soluble titanium, zirconium and tin compounds by converting these into the corresponding oxides.

By converting soluble initial substances into dilute solution for example, treating them with substances which are capable of precipitating the dissolved compounds in the form of oxide (in the form of hydroxide) and heating the precipitated oxide compounds there are obtained products of more or less compact and heavy nature which consequently have only a slight suitability for turbidity purposes or are not suitable or are only slightly suitable for colour pigments.

It has been suggested previously to prepare titanium complexes from materials wholly or partly consisting of titanium by heating these materials with sulphuric acid, in some cases under pressure, and treating the resulting mixtures with water and/or hydrofluoric acid and heat, whereby titanium compounds are precipitated by hydrolysis and the sulphuric acid and other impurities remain in solution. It has also been suggested previously to produce pure tin oxide from solutions of stannates by precipitating the tin in the form of oxide, washing the precipitate, calcining the washed precipitate and again washing with dilute acid or ammonium salt solutions.

According to the present invention a process for the production of highly dispersive metal oxides, capable of being used as pigments or turbidity agents, is characterised in that soluble or vaporisable compounds of titanium, zirconium

and tin are converted in their normal, solid or liquid, or in the gaseous condition with substances which are capable of converting them rapidly and as completely as possible into the form of hydroxide or oxide, as for example alkaline lyes, as for example caustic soda, ammonia or water in the form of steam, preferably superheated steam, the conversion products being heated to suitable temperatures, in the case of pigments for example to about 500 to 800° C, in the case of turbidity agents for example to about 900 to 1500° C, for the purpose of converting them into a voluminous form.

If the initial substances are in the form of solid bodies, as for example zirconium oxychloride, they may be subjected to the conversion directly in this form. Initial substances which normally exist in liquid form, as for example titanium chloride, can be treated in the liquid condition or in the form of vapour.

In carrying the invention into effect the procedure for example may be such that the initial substances are brought in the solid, liquid or vaporous condition into intimate contact with the conversion agents, as for example strong ammonia lye, superheated steam and the like, for example with the co-operation of agitating devices or the like. At first the oxides or hydroxides are in general obtained in more or less compact or granular form. By simply heating the conversion products to suitable temperatures these pass in a surprising manner into the desired finely divided voluminous form.

The temperatures of heating depend upon the particular purpose of the products. When manufacturing pigments for paints there are generally used temperatures of from 500 to 800° C preferably between 600 to 800° C. In the manufacture of turbidity media there are generally used temperatures about 900° C for example from 1000 to 1500° C, preferably between 1200 to 1500° C. In some cases the above mentioned temperature limits may not be reached or may be exceeded.

By mixing titanium tetrachloride in the liquid or vaporous state with concen-

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trated ammonia solution there is first obtained for example compact titanium hydroxide. When heating this to temperatures of for example 600 to 800° C it passes into a finely divided voluminous titanium oxide which constitutes an excellent pigment for paints.

By treating for example zirconium oxychloride with a strong ammonia solution and stirring there is first obtained granular zirconium hydroxide. When heated to temperatures of for example 1200 to 1500° C it is changed into a finely divided voluminous zirconium oxide which is an excellent turbidity medium for enamels or the like.

Volatile initial substances may preferably be treated in the vaporous state, for example by treating zirconium tetrachloride with superheated steam. By heating the hydroxide which is formed to temperatures of 1000° C and above there is obtained an excellent turbidity agent in voluminous form.

According to one method of carrying the invention into effect the conversion of the initial substances is effected at high temperatures, for example vaporous zirconium compounds are treated with superheated steam at temperatures of for example 600° C. With this method of operation for obtaining paint pigments subsequent heating of the product obtained may be omitted or the subsequent heating may be shortened. It is also possible if desired to have a subsequent heating, preferably at increased temperatures. When turbidity agents are to be obtained a subsequent heating to the required temperatures, for example from 1000 to 1400° C. is advisable.

It has been found of advantage to effect the subsequent heating directly after the conversion process. The annealing of the oxides may, however, be effected at a later period.

It has been finally found that the process can also be carried out in the presence of restricted quantities of solvents, for example, by subjecting the soluble initial substances in the form of concentrated or saturated solutions to the conversion process and converting the products obtained, as previously described, by heating to the voluminous condition. When operating in this manner it will be understood that the initial substances must not be diluted to such an extent as to form compounds, which on subsequent heating will not form finely divided voluminous products.

According to the invention it is also possible to produce preparations which are capable of producing excellent covering opacity with a simultaneous cloudy or

misty effect and which are characterised by their great resistance to vitrification and dissolving by the melt. When using the products as paint pigments they are distinguished by their great pliability.

EXAMPLES.

1. 5 kg. of titanium tetrachloride are evaporated and the vapours are heated in a porcelain tube to temperatures of about 500° C. At the end of the tube the vapours are brought in contact with superheated steam of about 500° C. By this operation the titanium chloride is almost instantaneously decomposed, the titanium being converted into oxide form, mainly into titanium oxide. The higher the temperature which is used the smaller is the content of titanium hydroxide.

The conversion product is washed with water, dried and then heated to about 600 to 800° C. The product obtained is very voluminous and constitutes an excellent pigment. Its apparent specific gravity only amounts to 0.65 whilst the apparent specific gravity of titanium oxide, which was obtained by the method hitherto usual, amounted to about 1.0.

2. 7.5 litres of concentrated ammonia solution (specific gravity 0.91) are mixed with 15 kg. of zirconium oxychloride whilst stirring. After conversion there are added 3 to 5 litres of water. The pasty mass is distributed in about 100 litres of water whereby the precipitate settles very quickly. The precipitate is then freed from ammonium by decantation, freed from ammonium chloride by water, filtered, dried and heated to a temperature of from 1200 to 1400° C. There is then obtained a highly voluminous excellent turbidity agent.

Instead of treating solid zirconium oxychloride this may also be treated in the presence of limited quantities of solvents, such as a highly concentrated or saturated solution in accordance with the preceding example.

3. Tin chloride (SnCl_4) having a boiling point of 112° C is evaporated. The vapour is heated to about 300° C in a porcelain tube. The heated tin chloride vapour is brought into contact with superheated steam. The conversion product is washed with water until the hydrochloric acid has been removed, is filtered and dried and then heated to a temperature which is somewhat above 900° C. There is obtained a highly voluminous excellent turbidity agent.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. A process for the production of

highly dispersive metal oxides capable of being used as pigments or turbidity agents, characterised in that soluble compounds of titanium, zirconium, and tin are converted in their normal, for example solid or liquid condition, with substances which are capable of converting them rapidly and as completely as possible into the form of hydroxide or oxide, as for example alkaline lyes, steam and the like, and the conversion products are heated to suitable temperatures, in the case of pigments for example to about 500 to 800° C, in the case of turbidity agents for example to about 900 to 1500° C, for the purpose of converting them into a voluminous form.

2. A process according to claim 1, characterised in that the initial substances are treated in the presence of limited quantities of solvents which do not restrict the conversion of the conversion products into voluminous form by heating, for example in the form of concentrated or saturated solutions.

3. Process according to claim 1, characterised in that volatile substances in the form of vapour are caused to react with substances, which are capable of converting them into oxides, preferably with

superheated steam, and the conversion products are subjected to heating in accordance with claim 1.

4. Process according to claims 1 and 3, characterised in that the conversion of the initial substances into oxide form is effected at high temperatures for example those of about 600° C when subsequent heating may be omitted if desired.

5. Process according to claims 1 to 4, characterised in that the subsequent heating of the oxides obtained by the conversion directly follows the conversion process.

6. A process for the production of highly dispersive metal oxides capable of being used as pigment or turbidity agents, substantially as described.

Dated this 27th day of December, 1928.

For the Applicants:

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Reference has been directed, in pursuance of Section 7, Sub-section 4, of the Patents and Designs Acts, 1907 to 1928, to Specifications Nos. 296,730, 206,284, 203,352, and 28,565 of 1908.

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